

<p>98-090084/09</p> <p>A60 E11 (A26)</p> <p>TAKEDA CHEM IND LTD</p> <p>96.01.29 96JP-012933 (97.08.12) C07F 7/10, 7/18 // C08G 77/388</p> <p>Preparation of isocyanate compounds with alkoxy:silyl groups - comprises reacting alkoxy:silylalkyl primary amine with phosgene in inert organic solvent in presence of alkali or alkaline earth metal compound</p> <p>C98-030549</p>	<p>TAKE 96.01.29</p> <p>* JP 09208589-A</p> <p><u>USE</u> Used as silicon modifiers for organic compounds.</p>	<p><u>ADVANTAGE</u></p> <p>Hydrochloric acid generated during the reaction with phosgene is efficiently removed by low cost raw materials, and side reactions avoided even in a distillation step. The low temperature reaction step necessary in the prior art is avoided by using tertiary amines. (II) are produced in high yield.</p>	<p><u>PREFERRED PREPARATION</u></p> <p>The alkali metal or alkaline earth metal compound is weak acid salt or oxide (specifically, CaO). The inert organic solvent is a polar solvent.</p>	<p><u>EXAMPLE</u></p>
<p>$R_1-R_3 = \text{hydrocarbon group or alkoxy};$</p> <p>$R_4, R_5 = \text{hydrocarbon group, alkoxy or alkoxysiloxy};$</p> <p>$R_6 = 1-8C \text{ alkylene; and}$</p> <p>$n = \text{integer of } 0-8.$</p> <p>At least one of R_1-R_5 is alkoxy.</p>	<p>$R_2-Si(R_1)(R_3)-(OSi(R_4)(R_5))_n-R_6-NCO$ (II)</p> <p>$R_2-Si(R_1)(R_3)-(OSi(R_4)(R_5))_n-R_6-NH_2$ (I)</p>	<p>$R_1-R_3 = \text{hydrocarbon group or alkoxy};$</p> <p>$R_4, R_5 = \text{hydrocarbon group, alkoxy or alkoxysiloxy};$</p> <p>$R_6 = 1-8C \text{ alkylene; and}$</p> <p>$n = \text{integer of } 0-8.$</p> <p>At least one of R_1-R_5 is alkoxy.</p>	<p>A 500 ml flask with a reflux condenser, dropping funnel and stirrer contained a solution of 9.9 g phosgene in 300 g EtOAc, to which was added 16.8 g CaO. A solution of 22.1 g γ-</p> <p>JP 09208589-A+</p>	

triethoxysilylpropylamine in 70 g EtOAc at 0-10°C was dropwise added over 1 hour. The mixture was warmed to 50°C and matured for 1 hour. The resulting CaCl₂ and excess CaO were filtered off, and the filtrate after removal of EtOAc was distilled to give 23.5 g colourless clear liquid having b.pt. of 92-94°C/5.0 mmHg. Gas chromatography indicated the product was γ -triethoxysilyl propylisocyanate (yield: 95.1%; purity: 99.5%), (HW) (5pp052DwgNo.0/0)